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Development and Optimization of an Online SPE-HPLC-FD Method for Quantification of Fluoroquinolones in Wastewater Effluents

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Abstract: Fluoroquinolones are antimicrobial agents widely found in environmental matrices and extensively studied due to their persistence and implications for multi-resistant bacteria. The presence of fluoroquinolones in the environment is mainly due to the incapability of wastewater treatment plants (WWTPs) to completely remove those compounds. The amount of fluoroquinolones released through effluents depends on the type of treatment used by the WWTPs. So, accurate analytical methods to quantify those compounds on WWTPs process and in effluents are crucial. Solid phase extraction (SPE) coupled to liquid chromatography is a straightforward technique that provides analyte extraction, cleanup, separation and detection while providing a good reproducibility and efficiency. The purpose of this work was the establishment of a novel method for quantification of Ofloxacin, Norfloxacin, Ciprofloxacin and Moxifloxacin on WWTPs effluents using on-line SPE. Samples were injected directly on a restricted access material column LichroCart 25-4 Lichrospher® RP-18 ADS (25 µm) and then transferred to an analytical column Luna PFP (2) (150 x 4.6 mm ID, 100 Å, 3 µm) for separation in isocratic mode with a mixture of 0.1% triethylamine in water (acidified to pH = 2.2 with trifluoroacetic acid) and ethanol as mobile phase; column oven was set at 45ºC. The detection was performed by fluorescence with an excitation wavelength of 290 nm and an emission wavelength of 460 nm. The injection volume of 100 µL of previous pre-concentrated sample was compared with larger volume injection of only filtered effluent samples. The study was conducted with effluent samples collected from a municipal WWTP in the north of Portugal.

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